

G. R. S. IX-5]

Previous references.

Int. 9-21

1921.

(Itc)

Later references.

File No. 55-21

Serial Nos. 1, (a) -

8a, 9-11

GENERAL AND REVENUE SECRETARIAT.

Serial Nos. 13

Part B for Jan 22-

SUBJECT.

Grant of leave to file a specification in respect of an invention for: Improvements in the trans formation of synthetic ammonia into a transportable product directly utilisable for agricultural use in conjunction with the production of carbonate of soda.

2067) No 3029 of 23-11-21 as thousandth
applicant to file a specification of
the invention referred to above

219) No 3497 of 10-1-22 notifying the filing
of the specification mentioned above

GENERAL AND REVENUE SECRETARIAT.

(etc)

NOTES.

File No. 63-21

FILE HEADING.

Invention for "Improvements in the transformation of Synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda".

From

Mr. W. Grant Rose. Solicitor. 2 Hastings Street Calcutta.

No.

Dated

Received 1.8.21

Diary No. 782

1 copy in duplicate
Enclos. 1 (power of atty)
(assignment)
check file

Serial No. 1

BRIEF ABSTRACT OF LETTER.

I am and an application for leave to file a specification on behalf of his clients L' Air Liquide Societe Anonyme. Pour l' Etude Et l' Exploitation Des Procédés Georges Claude in respect of an invention for "Improvements etc" refd to in the File Heading given above.

NOTES AND ORDERS.

L. R. R. Separately Subd.

Subd with necessary dfts for order.

19/8

Done
W. J. P.

Serial
No.

1A

To Secy office

421 & 15-8-21

Acknowledges receipt of Rs 10/-

2

To W. Grant Rose Secy

1265 & 26-8-21

Acknowledges receipt of W. Appl.

Returned to him as & states that the appl is
is receiving attention.

3

To The Supt of Stamps

1266 & 26-8-21

Forwards to him of attorney's
the deed of assignment for being impressed
with stamp.

4

To The Secy, Bd of R. Advice

No 1267 & 26-8-21

Forwards W. Appl for opinion.

5

From W. Supt of Stamps

86 & 31-8-21

Returns W. documents (Rs) duly
stamped.

6

From the Secy, Bd of R. Advice

No 18 & 29-10-21

Returns W. application & states
that leave may be granted to file the
specification.

Submitted into a deop.

AKS

7-11-21

but

7

To W. Grant Rose, Secy

No 3029 & 23-11-21

Authorizes W. filing of W. Specification

Recd by post

7/8/21-22

3573

*Ans 782
ge 4
2-8-21*

From

Mr. W. Grant Rose
Solicitor

Ho. 55-21-1

2 Hastings Street
Calcutta.

GEN & REV. SECTT
J & C
1 AUG 1921

To

The Chief Secretary to the Government of
His Highness the Maharaja of Mysore
General and Revenue Secretariat
Bangalore.

Sir,

I beg to enclose herewith the necessary papers in respect of an application for leave to file a specification on behalf of my clients L'Air Liquide Societe Anonyme Pour L'Etude Et L'Exploitation Des Procedes Georges Claude in respect of an invention for "Improvements in the transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda."

The following papers are enclosed herewith namely :-

- (1) Application in duplicate
- (2) Power of attorney
- (3) Assignment
- (4) Specification in duplicate

I am sending you a money order for Rs.12/- of which Rs.11/- is for the Mysore Government Stamp on the Power of attorney and Rs.1/- for the Mysore Government Stamp on the assignment and Rs.10/- being the prescribed fee for the application.

I may mention that the present application is based on my clients Indian application No.7051 of 1921 which was
filed

(6)

G.P.S. 56.]

GENERAL AND REVENUE SECRETARIAT

List of "B" Abstracts and Index Slips sent to the Press on the _____ 190 .

Branch.	Particulars of the papers despatched.	Remarks
Coupon. The remitter may write here any communication to the payee. Rs. 12/- As. Amount remitted in respect of Patent application to Dewan of Mysore on behalf of L'Air Leguide etc corresponding to Indian Patent application No 7051 of 1921. To J. W. Grant. Rose, Solicitor Remitter. 20/1/22 Hastings Street, Calcutta		

9-21

7855-21-1A

7855-21

7

Duplicate.

(To be returned to the General and Revenue Secretariat for being placed in the file.)

1. No. 421
2. Date of Remittance. 15/8/21
3. From whom received. Mr. W. Grant
Col. Solicitor & Attorneys
Street, Calcutta.
4. Date of receipt of amount in the General and Revenue Secretariat. 1-8-21
5. On what account. Appn Fee in
respect of 902-21-22
6. Amount (in figures and words). 10/-
rupees ten only
7. The head to which it should be credited.

(To be filled in by the Treasury.)

W. S. Chandra
Secretary to Government,
General Department.
AUG 15 1921
* The above amount has been duly received
and credited in the Treasury on this date.

Date

am
Hd. Acctt.

W. S. Chandra
Treasurer.

Try. Officer.

N. B.—Both the counterfoil and the duplicate to be returned to the General and Revenue Secretariat duly signed by the Treasury Department.

G. R. S. VI—G/32]

SUBJECT.....

Diary No. of letter replied to

General and Revenue Secretariat

Date of issue

Signed by

Section

File No.

Serial No.

No.

To

in Ltr. refce to your letter
of forwarding an application
for leave to file a specification
on behalf of your Clients
L'Air Liquide Societe Anonyme
Pour L'Etude Et L'Exploitation
Des Procedes Georges Claude
in respect of an invention
over.

24/8

Improvements in the transformation
of Synthetic ammonia into
a trans ~~portable~~ product directly
utilisable for agriculture in
conjunction with the production
of "Carbonates of Soda",
I am directed to ~~acknowledge~~ ^{return}
herewith ^{the excess amount of} half a rupee ^{out} of the
Sum of Rs 12/- received from you
to acknowledge the Sum of Rs 11/8 — only.

I am also to add That
your application is receiving
the attention of your
Shaweeh.

10/8/74
For Secy to Govt
19/8/74
10/8/74

List of enclosures.

Copied by

Examined by

DRAFT.

SUBJECT

17/8

Diary No. of letter replied to.....

GENERAL AND REVENUE SECRETARIAT,

, the.....191

Date of issue.....26/8/21

Signed by.....Mr. N. S. R.

Section. *Itc*
File No. *53-21*
Serial No. *4*
No. *LC 1267/Encls 2*

To

The Secy. Bd of
Ac. Advice
Bangalore

Has the honor to forward herewith

for report under Sec. 6, Cl. (2) of the

Mysore Inventions and Designs Regula-

tion, XI of 1894, an application for leave

to file a specification of an invention for

Improvements in the
transformation of synthetic
soda ammonia into a
transportable product
directly utilisable for agric.
By Order,*M. S. R.*10-8-21
To Secretary to Government.
2nd Dept.*In conjunction with the*
production of carbonate
of soda!! Recd from
Mrs. N. Grant Rose Solicitor
2. Hastings Street
Calcutta on behalf of
his clients L' Air Liquide
Societe Anonyme pour
L'Etude Et L'Exploitation
des Procédes Georges Claude 748, Rue St Lazare
Paris.

2

List of enclosures.

Copied by.....

Examined by.....

9-21

91c 55-21-5

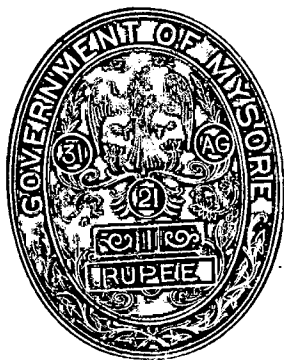
12

Copy of letter No. 86 dated 31st August 1921 from the
Superintendent of Stamps in Mysore, Bangalore to the Secretary to
Government, General Department.

I have the honour to forward herewith duly stamped eight Powers of
Attorney and eight Deeds of Assignment, forwarded with your ~~xxx~~ numbers
noted in the margin and to request the favour of an acknowledgment.
No. 1266, 1270, 1272, 1275, 1279, 1282, 1285, 1287. dated the 26th August 1921

Acknowledged vide no 1553 of 8-9-21,
.....

POWER OF ATTORNEY.



IN THE MATTER of the Inventions and Designs Regulation, 1894, of the Government of Mysore and in the matter of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE, of 48, Rue St Lazare, Paris, in the Republic of France.

We, the abovenamed, L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE, do hereby retain, constitute and appoint *William Grant Rose of No 2 Hastings Street in the Town of Calcutta Solicitor and Patent Agent* as our Agent and Attorney to apply for and obtain from the Government of Mysore and keep in force an Exclusive Privilege or Letters Patent for "Improvements in the transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda" and we authorise *him* to sign our name to such papers and writings and do such acts regarding the same, including the appointment of a substitute or substitutes, as may be necessary or expedient.

Dated this *22nd* day of *April* 1921.

THE COMMON SEAL of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE was hereto affixed in the presence of:-



"L'AIR LIQUIDE"
Societe Anonyme pour l'Etude et l'Exploitation des Procédés G. Claude
Un Directeur.

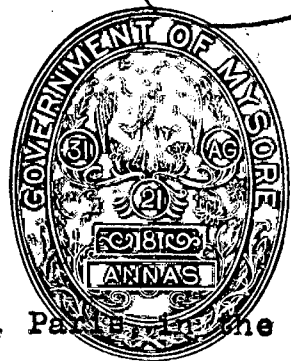
W. Grant Rose



7051 of 1921

u

A S S I G N M E N T.



WHEREAS I, GEORGES CLAUDE, of 48, Rue St Lazare, Paris, in the Republic of France, Engineer, am in possession of an Invention for "Improvements in the transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda"

Now therefore know ye that for and in consideration of the sum of five shillings to me in hand paid, the receipt of which is hereby acknowledged, I the said GEORGES CLAUDE, have assigned and transferred and by these presents do assign and transfer unto L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDES GEORGES CLAUDE, of 48, Rue St Lazare, Paris, in the Republic of France, their successors or Assigns, (hereinafter called "the said Assignee") the whole right, title and interest in and to Letters Patent to be obtained for the said Invention from the Government of Mysore the same to be held and enjoyed by the said Assignee to the end of the term or period for which such Letters Patent may be granted, or any prolongation or extension thereof, as fully and entirely as the same could have been held and enjoyed by me had this Assignment not been made.

IN WITNESS whereof I, the said GEORGES CLAUDE, have hereunto set my hand and seal this 22nd day of April 1921.



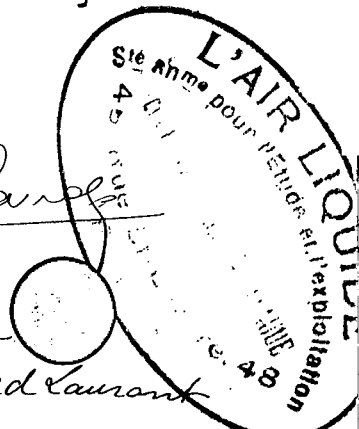
SIGNED, SEALED AND DELIVERED

at Paris

in the presence of:-

*Pierre Hauquier, Engineer at L'air Liquide
residing in Boulogne-sur-Mer, 34 Rue Alfred Laurant*

*Gaston Scala Engineer at L'air Liquide
residing in Paris 19 Rue Baum*



15-

copy of letter no 18 of 29-10-21 from the Dept.
of the advice to the High Court, G. Govt.

I am directed to ~~forward~~ return herewith
the following 9 applications & to state that leave maybe
granted to file the specification of inventions
therein contained.

1 x x y.

4. For transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda.

5 x x x

Indian Appln
7051 of 1921

PAPERS FOR APPLICATION FOR

LETTERS PATENT

IN

M Y S O R E

L'AIR LIQUIDE, SOCIÉTÉ ANONYME POUR L'ÉTUDE ET
L'EXPLOITATION DES PROCÉDÉS GEORGES CLAUDE.

(17)

A P P L I C A T I O N .

TO THE DEWAN OF MYSORE.

THE APPLICATION of L'AIR LIQUIDE,
SOCIETE ANONYME POUR L'ETUDE ET
L'EXPLOITATION DES PROCEDES GEORGES
CLAUDE, of 48, Rue St Lazare, Paris,
in the Republic of France, for leave
to file a Specification under Part 1
of the Mysore Inventions and Designs
Regulation, 1894.

1. The Applicants are in possession of an Invention for
"Improvements in the transformation of synthetic ammonia into
a transportable product directly utilisable for agriculture in
conjunction with the production of carbonate of soda"
they are the Assignees of the Inventor
thereof; and, to the best of their information and belief, the
Invention is new within the meaning of Part I of the Inven-
tions and Designs Regulation, 1894, and no circumstance exists
which, if the Applicants are authorised to file a specification
and file it in accordance with that Part, will disentitle
them to an Exclusive Privilege thereunder in respect of the
Invention.

The Applicants have applied for a Patent in British India
in respect of this Invention No. ~~5896~~ dated the 10th day of

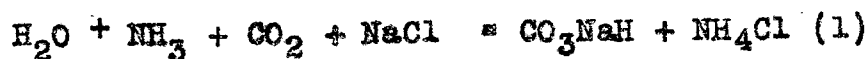
~~July, 1920.~~ 7051 of 1921 dated the 3rd June 1921

2. The following is a description of the invention:-

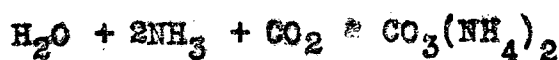
LALSA
P.L.E.E
L.E.D.P
S.C
W.M.

In our British Specification No. 130,365 of the 29th January, 1918, a process was described for the manufacture of ammonium chloride and carbonate of soda, both in a solid state, which utilises as raw materials; sea salt, water, coal and air. It was mentioned that in order to obtain solid ammonium chloride, it was necessary to concentrate by evaporation the ammonium chloride liquor obtained or to separate from it by fractional crystallisations the carbonate of soda that it contains.

The object of the present invention is to obviate these evaporations or crystallisations, which consume heat, and to increase the purity of the solid chloride. To this end it modifies the process indicated in Schreib's German Patent No. 36,003 of the 14th June 1885. According to this patent, when the precipitation of the bicarbonate of soda has taken place in accordance with the equation:-



and the liquid containing the ammonium chloride mixed with bicarbonate of soda has been separated, sea salt is added to the liquid up to saturation point, then ammonia, and then carbon dioxide, so as to form neutral carbonate of ammonia, according to the reaction:-



This is cooled to about 5°C. Almost the whole of the ammonium chloride is precipitated and is collected so as to be centrifuged. Sea salt is then dissolved again in the

carried out as in the Schreib process, but the solid ammonium chloride is in this way obtained in a higher state of purity

The average figures resulting from putting the preceding operations into practice will be indicated hereunder by way of example:-

The liquor from which the bicarbonate of soda has been separated has the following composition:-

NH_4Cl = 225 kilogrammes per cubic metre of liquor

NaCl = 100 kilogrammes per cubic metre of liquor

N H_3 = 15 kilogrammes per cubic metre of liquor

CO_2 = 31 kilogrammes per cubic metre of liquor

Density: 18 degrees Baume.

This liquor is passed into a vat with a stirrer and a cooling coil. While the stirring is going on we add.

Sea salt: 100 kilogrammes per cubic metre of the original liquor

N H_3 : 62 kilogrammes per cubic metre of the original liquor

CO_2 62 kilogrammes per cubic metre of the original liquor

The stirrer is stopped and the contents of the vat cooled to 5° Centigrade. The ammonium chloride is precipitated and is centrifuged and then stove-dried at not less than 60° C.

The mother liquors resulting from the treatment have the same volume as the original liquor and have the following composition:-

$\text{N H}_4\text{Cl}$: 75 kilogrammes per cubic metre of the original liquor.

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NaCl: 200 kilogrammes per cubic metre of the original liquor

$\text{CO}_3(\text{N H}_4)_2$: 191 kilogrammes per cubic metre of the original liquor

They are passed into a tank provided with a stirrer in which they are diluted with 150 litres of water per cubic metre of the original liquor and then 90 kilogrammes of sea salt per cubic metre of this original liquor are dissolved therein. Carbon dioxide is passed into it in order to precipitate the bicarbonate of soda. The latter is separated washed and filtered and the mother liquor is again treated as above.

However, the chloride obtained still contains, even after centrifugation, alkaline mother liquors, on account of the presence of a small quantity of dissolved carbonates of soda and this alkalinity would be objectionable when employing the chloride as manure. In order to get rid of it, according to the present invention, the impure solid ammonium chloride obtained is heated to a temperature in the neighbourhood of 70°C . At this temperature the latter reacts on the small quantity of carbonates of soda of the mother liquors, giving carbonate of ammonia. The latter, on account of its volatility is eliminated by heat together with what was present in the mother liquors. The ammonium chloride remaining is then perfectly neutral and does not contain more than from 1 to 3% of sodium chloride, a quantity which is insufficient to impair the quality of the product.

After a certain number of precipitations of bicarbonate of soda and ammonium chloride, according to the Schreib pro-

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subsequent cooling of the solution of ammonium chloride and ammonium carbonate free from bicarbonate, with a view to bringing about the precipitation of the ammonium chloride.

Consequently, according to the present invention, the liquid ammonia is evaporated for example in a coil in indirect contact with the ammonium chloride solution to be cooled, and the gaseous ammonia resulting therefrom is passed into a tank containing the brine cooled by a circulation of water, in which tank the dissolving and the carbonating are effected. It will be possible, if occasion arises on account of a higher surrounding temperature, to increase the quantity of cold available by utilising the expansion with external work of the gaseous ammonia. To this end the liquid ammonia is evaporated under pressure and the gas resulting therefrom is caused to expand in a motor which increases the quantity of cold by a quantity corresponding to the whole of the mechanical work produced. From there, this gas flows in indirect contact with the liquid to be cooled.

A similar remark might be made with regard to the use of the carbon dioxide intended for the precipitation of the carbonate of soda, for this substance can be produced in a solid or liquid state by the very process of manufacturing the hydrogen intended for the synthesis of ammonia, as was indicated in our British Specification No. 130,365 referred to above.

24

HAVING NOW particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, we declare that what we claim is:-

1. Improvements in the process of transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda which formed the subject matter of our prior Specification No. 130,365 consisting in the combination of the following steps:

(a) in adding to the liquor which has precipitated bicarbonate of soda, sea salt, ammonia and carbon dioxide the sea salt added being in such limited quantity that it always remains dissolved throughout the subsequent operation and consequently does not mix with the ammonium chloride.

(b) cooling the solution to about 0° to 5°C.

(c) eliminating the ammonium chloride precipitated.

(d) treating the remaining liquor with sea salt and carbon dioxide with precipitation of bicarbonate of soda and formation of a fresh ammonium chloride liquor.

(e) centrifuging the ammonium chloride obtained and heating it to a temperature in the neighbourhood of 70°C. in order to destroy the alkalinity thereof, all substantially as hereinbefore described.

2. The process as claimed in claim 1 wherein the evaporation of the ammonia is utilised for cooling the solution which is to deposit the chloride of ammonia and in sending the ammonia thus vaporised into the solution in which it

26

3. The Applicants, therefore, apply for leave to file a Specification of the Invention pursuant to Part I of the Inventions and Designs Regulation, 1894.

THE COMMON SEAL of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDÉS GEORGES CLAUDE was hereto affixed in the presence of:-



"L'AIR LIQUIDE"
Société pour l'Étude et l'Exploitation des Procédés G. Claude
Un Directeur
Georges Claude

We, the Applicants abovenamed, do declare that the facts stated in this Application are true to our knowledge, except as to matters stated on information and belief and as to those matters we believe them to be true.

Dated this 29th day of April 1926.

THE COMMON SEAL of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCÉDÉS GEORGES CLAUDE was hereto affixed in the presence of:-



"L'AIR LIQUIDE"
Société pour l'Étude et l'Exploitation des Procédés G. Claude
Un Directeur
Georges Claude

27929

OFF. & REV. SECT.
14 DEC 1921

From

Mr. W. Grant Rose

Solicitor.

No. 2 Hastings Street.

Calcutta.

To

The Chief Secretary to the Government of

His Highness the Maharajah of Mysore

General Department Bangalore

Mysore.

Calcutta 10th December 1921.

Sir,

Patent Register No 9 of 1921/22.

16/12
With reference to your letter No I C 3036/I & C60-21-9 dated the 23rd ultimo forwarding the Government order granting leave to file Specification in respect of the application of L'Air Liquid &c being Register No 9 of 1921/22 I beg to enclose herewith under registered cover two copies of the specification in respect of such application which together with the two copies of specification already submitted with the application will make four copies thereof and I beg that the same may be filed in terms of the order granting leave

I also beg to enclose herewith the Government Order No 3029/14C 55-21-7 dated the 23rd November 1921 so that the certificate at foot thereof certifying that the specification has been filed and the fees paid may be duly signed and the order thus completed returned to me.

I am

20

I am remitting by money order Rs.30/- being the prescribed fee for filing the specification herein,

Kindly acknowledge receipt of the papers enclosed herewith and the remittance sent.

I have the honour to be,

Sir,

Your most obedient servant.

W Grant Jones

Enclos

9-21
31
The 55-21-8a D. 2935

Duplicate.

(To be returned to the General and Revenue Secretariat for being placed in the file.)

1. No. 468
2. Date of Remittance 6-1-22
3. From whom received Mr. W. Grant
Roh. Collector, Calcutta
4. Date of receipt of amount in the General and Revenue Secretariat 16-12-21
5. On what account Police fee in
respect of F.M. no. 7 of 21-22
6. Amount (in figures and words) Rs. 30/-
Rupees thirty only
7. The head to which it should be credited.

(To be filled in by the Treasury)

M. Appanibh...
For Secretary to Government,
As General Department.

The above amount has been duly received
and credited in the Treasury on this date.

Date

8/1/22

Hd. Acctt.

Treasurer.

Try. Officer.

N. B.—Both the counterfoil and the duplicate to be
returned to the General and Revenue Secretariat
duly signed by the Treasury Department.

abz 9221 ✓
94c 55-21-9
32

GOVERNMENT OF HIS HIGHNESS THE MAHARAJA OF MYSORE,
GENERAL AND REVENUE SECRETARIAT

Bangalore, Dated 10/1/1922

No. LC3797

LC55-21-9

NOTIFICATION.

(PATENT)

The following specification of an invention has been filed in accordance with Sections 8, 9, 10 and 11 of Regulation XI of 1894 (The inventions and Designs Regulations) and is open to inspection at the Mysore Government Secretariat on application to the ~~Chief~~ ^{General} Secretary to the Government of His Highness the Maharaja of Mysore, and on payment of a fee of one Rupee.

By L'Air Liquide, Societe Anonyme Pour L'etude et L'exploitation des procedes Georges Claude, of 48, Rue St Lazare, Paris.

" Improvements in the transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda.

By Order,

M. Appannappa
22-1-22
For Chief Secretary to Government,
Glasgow
mc

To

The Compiler, Mysore Gazette, for publication.

SUBJECT.....

Diary No. of letter replied to.....

Section 9tc

File No. 55-21

Serial No. 11

General and Revenue Secretariat

()

, the

191..

No. 3799-801 / Enclure
to each

To

- 1) The Supdt, Govt Museum
- 2) The Chief Engineer of Mysore
- 3) The Secy Bd of Education.

Date of issue

10/1/22

Signed by

Mr J AG

Has the honor to forward
herewith for record in his office copy
of specification of an invention "for
"Improvements in the transformation of
synthetic ammonia into a transportable
product directly utilisable for —
agriculture in conjunction with the
production of carbonate of soda" by
Léon. Liquide, Société Anonyme Pour
L'étude Et L'Exploitation des
procédés Georges Claude, of 48, Rue
over.

1.20

St Lazare, Paris.

- D.O.

~~Aug 12 2~~
- For keep help of gloys

incl

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List of enclosures.

Copied by

Examined by

A P P L I C A T I O N .

TO THE DEWAN OF MYSORE.

THE APPLICATION of L'AIR LIQUIDE,
SOCIETE ANONYME POUR L'ETUDE ET
L'EXPLOITATION DES PROCEDES GEORGES
CLAUDE, of 48, Rue St Lazare, Paris,
in the Republic of France, for leave
to file a Specification under Part 1
of the Mysore Inventions and Designs
Regulation, 1894.

1. The Applicants are in possession of an Invention for
"Improvements in the transformation of synthetic ammonia into
a transportable product directly utilisable for agriculture in
conjunction with the production of carbonate of soda"

they are the Assignees of the Inventor
thereof; and, to the best of their information and belief, the
Invention is new within the meaning of Part I of the Inven-
tions and Designs Regulation, 1894, and no circumstance exists
which, if the Applicants are authorised to file a specification
and file it in accordance with that Part, will disentitle
them to an Exclusive Privilege thereunder in respect of the
Invention.

The Applicants have applied for a Patent in British India

in respect of this Invention No. ~~5896~~ dated the 10th day of

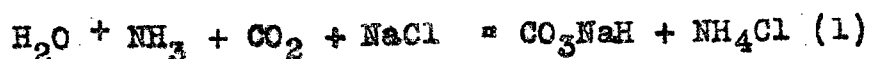
~~July, 1920.~~ 7051 of 1921 dated 3rd June 1921

2. The following is a description of the invention:

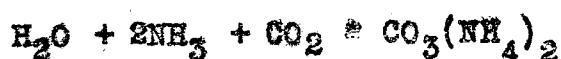
*L. a L. S. a. p. s.
L. S. S. L. E. D.
P. H. C. W. M.*

In our British Specification No. 130,365 of the 29th January, 1918, a process was described for the manufacture of ammonium chloride and carbonate of soda, both in a solid state, which utilises as raw materials; sea salt, water, coal and air. It was mentioned that in order to obtain solid ammonium chloride, it was necessary to concentrate by evaporation the ammonium chloride liquor obtained or to separate from it by fractional crystallisations the carbonate of soda that it contains.

The object of the present invention is to obviate these evaporations or crystallisations, which consume heat, and to increase the purity of the solid chloride. To this end it modifies the process indicated in Schreib's German Patent No. 36,003 of the 14th June 1885. According to this patent, when the precipitation of the bicarbonate of soda has taken place in accordance with the equation:-

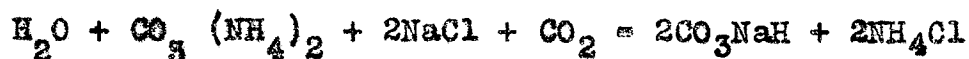


and the liquid containing the ammonium chloride mixed with bicarbonate of soda has been separated, sea salt is added to the liquid up to saturation point, then ammonia, and then carbon dioxide, so as to form neutral carbonate of ammonia, according to the reaction:-



This is cooled to about 5°C. Almost the whole of the ammonium chloride is precipitated and is collected so as to be centrifuged. Sea salt is then dissolved again in the

remaining liquid and carbon dioxide is then passed into it. Bicarbonate of soda is formed which precipitates and again we have ammonium chloride according to the reaction:-



We therefore find ourselves in the presence of the same substances as those obtained in reaction (1) and upon them the same series of operations is commenced as above. Under these conditions it will be seen that a given quantity of the original liquid can furnish in succession without evaporation very large quantities of bicarbonate of soda and ammonium chloride, both in a solid state.

Now by operating in this way, the two following disadvantages are met with: firstly, when, in order to saturate with sea salt the liquid containing the ammonium chloride the latter is passed, according to Schreib, over a mass of solid salt, part of the ammonium chloride is deposited on the salt. Moreover when carbon dioxide is subsequently passed for the purpose of forming neutral carbonate of ammonia, part of the dissolved sea salt is precipitated with the ammonium chloride, thereby diminishing its purity.

The object of the present invention is to remedy these two defects. With this aim in view, instead of adding the salt to the liquor up to saturation point we limit ourselves to the quantity that is necessary in order that it may remain dissolved throughout the duration of the subsequent operations. In this way it will not become mixed with the solid ammonium chloride. The other operations are then

carried out as in the Schreib process, but the solid ammonium chloride is in this way obtained in a higher state of purity

The average figures resulting from putting the preceding operations into practice will be indicated hereunder by way of example:-

The liquor from which the bicarbonate of soda has been separated has the following composition:-

NH_4Cl = 225 kilogrammes per cubic metre of liquor
 NaCl = 100 kilogrammes per cubic metre of liquor
 N H_3 = 15 kilogrammes per cubic metre of liquor
 CO_2 = 31 kilogrammes per cubic metre of liquor
Density: 18 degrees Baume.

This liquor is passed into a vat with a stirrer and a cooling coil. While the stirring is going on we add.

Sea salt: 100 kilogrammes per cubic metre of the original liquor

N H_3 : 62 kilogrammes per cubic metre of the original liquor

CO_2 62 kilogrammes per cubic metre of the original liquor

The stirrer is stopped and the contents of the vat cooled to 5° Centigrade. The ammonium chloride is precipitated and is centrifuged and then stove-dried at not less than 60° C.

The mother liquors resulting from the treatment have the same volume as the original liquor and have the following composition:-

$\text{N H}_4\text{Cl}$: 75 kilogrammes per cubic metre of the original liquor.

NaCl: 200 kilogrammes per cubic metre of the original liquor

$\text{CO}_3(\text{N H}_4)_2$: 191 kilogrammes per cubic metre of the original liquor

They are passed into a tank provided with a stirrer in which they are diluted with 150 litres of water per cubic metre of the original liquor and then 90 kilogrammes of sea salt per cubic metre of this original liquor are dissolved therein. Carbon dioxide is passed into it in order to precipitate the bicarbonate of soda. The latter is separated washed and filtered and the mother liquor is again treated as above.

However, the chloride obtained still contains, even after centrifugation, alkaline mother liquors, on account of the presence of a small quantity of dissolved carbonates of soda and this alkalinity would be objectionable when employing the chloride as manure. In order to get rid of it, according to the present invention, the impure solid ammonium chloride obtained is heated to a temperature in the neighbourhood of 70°C . At this temperature the latter reacts on the small quantity of carbonates of soda of the mother liquors, giving carbonate of ammonia. The latter, on account of its volatility is eliminated by heat together with what was present in the mother liquors. The ammonium chloride remaining is then perfectly neutral and does not contain more than from 1 to 3% of sodium chloride, a quantity which is insufficient to impair the quality of the product.

After a certain number of precipitations of bicarbonate of soda and ammonium chloride, according to the Schreib pro-

cess modified as above all the impurities in the sea salt employed, particularly the magnesium salts are concentrated in the ammonium chloride mother liquors. Since it is here a question of preparing manures it is desirable to bring these magnesium salts into a form useful for agricultural purposes. For this purpose a soluble phosphate is added to the ammoniacal liquor. The magnesium settles out in a state of ammonium-magnesium phosphate, which in this way is found mixed with the solid ammonium chloride.

Finally, the present invention relates to means for producing economically the cold necessary for effecting the precipitation of the ammonium chloride after the addition of the sea salt, the ammonia and the carbon dioxide. These means consist in utilising the cold of the liquid ammonia and of the solid or liquid carbon dioxide. In point of fact, when synthetic ammonia is obtained in the producing apparatus directly in the liquid form, its passage from the liquid state into the gaseous state produces a considerable quantity of cold. This cold would be badly utilised if the ammonia were passed in a liquid form into the liquor intended for carbonating, for it would only serve to hinder the rise of temperature due to the heat of dissolution of the ammonia in the liquid and to the heat of reaction by the carbon dioxide, which rise of temperature will be more rationally eliminated by a mere circulation of water keeping the reacting substances at a temperature in the neighbourhood of the surrounding temperature. The cold produced at a low temperature by the vaporisation of the ammonia finds its rational employment in the

subsequent cooling of the solution of ammonium chloride and ammonium carbonate free from bicarbonate, with a view to bringing about the precipitation of the ammonium chloride.

Consequently, according to the present invention, the liquid ammonia is evaporated for example in a coil in indirect contact with the ammonium chloride solution to be cooled, and the gaseous ammonia resulting therefrom is passed into a tank containing the brine cooled by a circulation of water, in which tank the dissolving and the carbonating are effected. It will be possible, if occasion arises on account of a higher surrounding temperature, to increase the quantity of cold available by utilising the expansion with external work of the gaseous ammonia. To this end the liquid ammonia is evaporated under pressure and the gas resulting therefrom is caused to expand in a motor which increases the quantity of cold by a quantity corresponding to the whole of the mechanical work produced. From there, this gas flows in indirect contact with the liquid to be cooled.

A similar remark might be made with regard to the use of the carbon dioxide intended for the precipitation of the carbonate of soda, for this substance can be produced in a solid or liquid state by the very process of manufacturing the hydrogen intended for the synthesis of ammonia, as was indicated in our British Specification No. 130,365 referred to above.

HAVING NOW particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, we declare that what we claim is:-

1. Improvements in the process of transformation of synthetic ammonia into a transportable product directly utilisable for agriculture in conjunction with the production of carbonate of soda which formed the subject matter of our prior Specification No. 130,365 consisting in the combination of the following steps:

(a) in adding to the liquor which has precipitated bicarbonate of soda, sea salt, ammonia and carbon dioxide the sea salt added being in such limited quantity that it always remains dissolved throughout the subsequent operation and consequently does not mix with the ammonium chloride.

(b) cooling the solution to about 0° to 5°C.

(c) eliminating the ammonium chloride precipitated.

(d) treating the remaining liquor with sea salt and carbon dioxide with precipitation of bicarbonate of soda and formation of a fresh ammonium chloride liquor.

(e) centrifuging the ammonium chloride obtained and heating it to a temperature in the neighbourhood of 70°C. in order to destroy the alkalinity thereof, all substantially as hereinbefore described.

2. The process as claimed in claim 1 wherein the evaporation of the ammonia is utilised for cooling the solution which is to deposit the chloride of ammonia and in sending the ammonia thus vaporised into the solution in which it

is absorbed, the heat produced by this latter reaction being eliminated by a circulation of water.

3. In the process claimed in claim 1 utilising in a similar manner as specified in claim 2 the evaporation of the liquid or solid carbonic acid.

4. The process claimed in claims 1-3 wherein the quantity of cold available is increased if necessary by utilising the expansion with external work of the ammonia and of the carbonic acid.

5. The process of transformation of synthetic ammonia into a transportable product directly utilisable in agriculture in conjunction with the production of carbonate of soda substantially as hereinbefore described.

3. The Applicants, therefore, apply for leave to file a Specification of the Invention pursuant to Part I of the Inventions and Designs Regulation, 1894.

THE COMMON SEAL of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE was hereto affixed in the presence of:-

"L'AIR LIQUIDE"
Société pour l'Etude et l'Exploitation des Procédés G. Claude
Un Directeur,
Georges Claude



We, the Applicants abovenamed, do declare that the facts stated in this Application are true to our knowledge, except as to matters stated on information and belief and as to those matters we believe them to be true.

Dated this 22nd day of April 1920.

THE COMMON SEAL of L'AIR LIQUIDE, SOCIETE ANONYME POUR L'ETUDE ET L'EXPLOITATION DES PROCEDES GEORGES CLAUDE was hereto affixed in the presence of:-

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Un Directeur,
Georges Claude

